



Conductivity Measurement of ZnO Nanowires Using the Powder-Solution-Composite Technique

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The powder solution composite (PSC) method was used to obtain the conductivity of ZnO nanowires from impedance spectroscopy measurements of ZnO nanowires dispersed in solution. The PSC method was calibrated by comparing impedance spectroscopy measurements of ZnO powder dispersed in solution with four-point probe measurements on bulk ZnO ceramic pellets. Conductivity values for ZnO nanowires obtained using this novel approach were found to be comparable to reported values obtained using two-point and four-point probe measurements of single or multiple nanowire devices isolated on a substrate. It is shown that application of the PSC method to ZnO nanowire solutions avoids the difficult process of forming electrical contacts to nanowires and allows for the non-destructive in-situ measurement of the average electrical properties of a large quantity of nanowires.

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Determining the average conductivity of large numbers of quasi one dimensional structures such as nanowires (NWs) and carbon nanotubes remains a challenge. The most straightforward method of determining the conductivity of NWs is by running a series of I-V measurements on a NW device with two or more electrodes. However, application of electrical contacts to NWs is itself challenging. NW devices are still most often constructed via a "pick and place" type approach in which NWs in solution are randomly dispersed onto a substrate. This approach requires either sophisticated or low yield processes to fabricate electrodes for individual or multiple NW devices and is not suitable for rapid assessment of the average conductivity of a large number of NWs. If instead pre-fabricated electrodes are used, it has been shown that bending of NWs over the edge of the electrodes¹ changes the strain on the NWs and affects the mobility and carrier concentration.² The resistivity of free standing NWs was recently determined without forming electrodes using a low energy electron point source microscope;³ however, this technique may be difficult to implement in a production environment and may only be used for measurement of individual NWs.

A method of measuring conductivity that circumvents the tedious process of nanowire electrode formation and also allows simultaneous measurement of a large number of NWs is critically needed. AC-impedance spectroscopy is a non-destructive, precise, and highly repeatable technique that has been used to characterize ceramic powder solution composites. The powder-solution-composite (PSC) method was recently developed as a method for combining AC-impedance spectroscopy measurements with effective medium theory to determine the conductivity of a loose ceramic powder.⁴ The PSC method is based on measuring the AC-impedance spectra of "slurry composites" made from mixing the ceramic powder of unknown conductivity in a series of aqueous electrolytes spanning an appropriate range of known conductivity. Although the possibility for measuring ellipsoid particles and high aspect ratio fibers has been demonstrated,^{5,6} there have been no reports utilizing the PSC method to measure the conductivity of NWs.

In this work, we use the PSC method to assess the average electronic properties of ZnO NWs in solution without fabrication or patterning of electrodes. The validity of the PSC method was first confirmed for powdered bulk ZnO ceramics via comparison to four-point probe measurements on ZnO sintered bars. The PSC method was then applied to determine the average conductivity of ZnO NWs suspended in NaCl solution. We demonstrate that the PSC technique can be used as a non-destructive technique for the charac-

terization of the average electrical properties of large numbers of NWs in solution.

Recently, AC dielectrophoresis, in which nanowires dispersed in solution are flowed across a substrate and aligned to lithographically defined electrodes with the aid of AC electric fields,^{7,8} has emerged as a promising method for NW integration. Before this method of NW integration can be scaled to production, techniques for rapidly assessing the quality of batches of nanowire solution will be required. This work shows that the PSC technique should be useful for in-situ assessment of the average quality of suspended NW solutions prior to their use on a device substrate.

Experimental

Sample preparation.—Bulk ceramic specimens were prepared from raw powders of ZnO (99.9%, Alfa Aesar) that were ball milled for 6 h to reduce the particle size and dried overnight. The powder was then calcined at 950°C for 3 h and then uniaxially pressed under 100 MPa into a 2.5 cm-diameter pellet. The bulk ZnO pellet was subsequently sintered at 1100°C for 4 h. X-ray diffraction was used to check the phase purity. The ceramic powder used in the resistivity measurements was obtained by pulverizing the sintered pellet by agate mortar and then filtrated with a 150 μm diameter sieve.

A modified method for synthesis and harvesting ZnO NWs has been formulated in this research. As opposed to use of a catalyst⁹ or ZnO seed layer,¹⁰ a dense layer of ZnO NWs was grown on a carbonized positive photoresist (Shipley S1818) nucleation layer^{11–14} using a conventional solid vapor transport method. First, a 2 μm thick layer of photoresist was spin-coated onto a 1 \times 1 (inch) (Ref. 2) Si substrate and then carbonized at 900°C for 60 min and 3 Torr under 150 sccm N₂ flow. A 1:1 mixture of pure ZnO (99.9%, Alfa Aesar) and graphite powders (99.9995%, Alfa Aesar) was then prepared and placed in a tube furnace at 925°C. A 150 sccm flow of N₂ was used to carry the Zn vapor (produced by carbothermal reduction⁹) downstream to the substrates at 770 °C. Oxygen gas at 1 sccm was introduced 5 cm from the samples to aid NW growth. After 3 h of growth, the substrate was quenched in air and a thick white layer of free-standing ZnO NWs (nano-carpet) was *peeled off* from the carbonized photoresist (see Fig. 1). A large quantity (~50 mg) of NWs was grown. The average length and diameter of 10 μm and 45 nm, respectively, were determined by using a dual beam field emission scanning electron microscope (Quanta 3D SEM, FEI). Harvesting of the NWs into solution was performed via sonication of the nano-carpet in de-ionized (DI) water for 1 min. Finally, as described in the next section, NaCl was dissolved into the NW/DI water suspension to form solutions over a wide range of conductivity.

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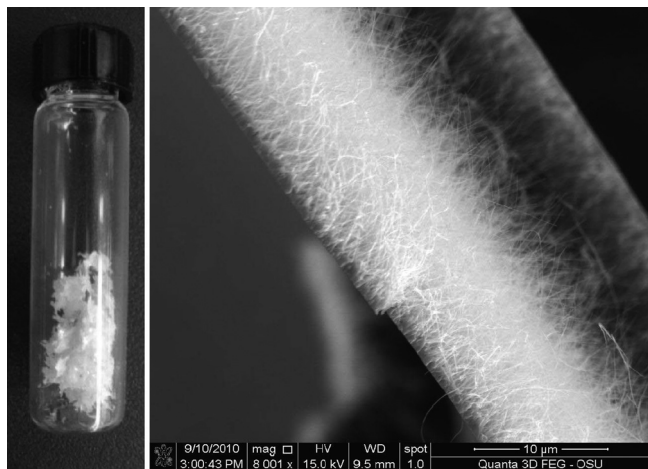


Figure 1. (Left) 50 mg of ZnO NWs grown via vapor solid transfer using carbonized photoresist as a nucleation layer, (right) peeled off sheet of free standing NW carpet.

Electrical measurements.—The bulk ceramic conductivity was obtained using a four-point DC conductivity measurement.¹⁵ The sintered pellet was cut into a rectangular bar [$10 \times 10 \times 5$ mm (Ref. 3)] with small notches made at approximately 1/3 and 2/3 positions along the length of the bar. The outer electrodes were made by applying silver paint at the both ends of the bar while the inner electrodes were made by wrapping two gold wires tightly and equally spaced around the marked notches.¹⁶ The four-point DC measurement was implemented by applying a current (Model 195A, Keithley) to the outer electrodes and measuring the voltage drop across the inner gold wires using a digital multi-meter (Fluke 179 multimeter, Optimum Energy Products Ltd.).

For the PSC measurements, a suspended NaCl solution of either pulverized ZnO powder or ZnO NWs was used. There are two primary concerns in preparing the suspensions. First, the solution must not react with the material under test.^{4,5} Second, in order to determine the conductivity of the material under test, it must be easy to adjust the conductivity of the solution over a range spanning the expected conductivity of the sample. NaCl has been shown to allow a wide range of solution conductivity, from 10^{-4} to 0.5 S cm^{-1} .⁴ A series of NaCl solutions with the conductivity ranging from 1.4×10^{-4} S cm^{-1} (0.001 M) to 2.8×10^{-1} S cm^{-1} (0.3 M) were prepared by dissolving NaCl (99.5%, Alfa Aesar) in DI water. Each of these solutions was then mixed with either pulverized ZnO powders of approximately 40% volume fraction or ZnO NWs of approximately 1% volume fraction.

Prepared solutions were placed into an apparatus consisting of a polyethylene tube (4.7 mm inner diameter \times 12.5 mm length) and two stainless steel electrodes.⁴ The electrodes were polished with silicon carbide abrasive paper (1200/P2500, Buehler company) until the surface appeared shiny and smooth. Note that attention should be paid to the electrode surface roughness since it has been shown to have an effect on the impedance spectra, especially at low frequencies.¹⁷ The inner electrode spacing in this experiment was 10 mm. A 1296 Hewlett Packard low-frequency impedance spectrometer in conjunction with a Solartron 1260 frequency analyzer was used for measuring the ac voltage response of the system. The frequency range used was from 10 Hz to 25 MHz. Simulations of impedance spectra were conducted using Zview Impedance Simulation software.

Results and Discussion

The PSC technique^{4-6,18} is based on impedance spectroscopy (IS) and effective medium theory. There are three basic requirements of the system to be analyzed: (1) a moderately conductive

matrix (in our case an NaCl solution), (2) highly conducting particles dispersed in the solution (here either ZnO powder or ZnO NWs), and (3) a relatively high impedance layer between the solution and particle which can either be an oxide shell, Schottky barrier formation, or, as is likely in our case, an electrical double layer. AC impedance spectra are typically plotted in the Nyquist representation (negative imaginary impedance vs. real impedance) with increasing frequency from right to left (frequency markers, included as small numbers along each plot, represent the logarithm of the frequency).

Shown in Fig. 2 is a Nyquist plot of simulated and experimental impedance measurements of 0.001 M NaCl with 40% volume fraction ZnO ceramic powder. (Due to the difficulties associated with obtaining four-point conductivity measurements for large numbers of NWs, the validity of the PSC method for ZnO was first confirmed by comparing the conductivity obtained using the PSC method on ZnO powders with the conductivity obtained using four-point measurements of ZnO ceramics.) Each electrical component in the system (e.g. electrode, solution/composite) is represented by a semi-circular arc in the plot.¹⁹ The presence of the high impedance layer between the particle and the solution is critical. The electrical double-layer creates a "frequency-switchable" interfacial impedance at the ZnO particle surface (described fully in Refs. 6 and 18) that results in the division of the solution impedance arc into two separate arcs on a Nyquist plot. As a result, two cusps are typically observed in impedance spectra for these types of systems. The cusp at low frequency excitation (denoted as R_{DC} on the right side of Fig. 2) occurs because the high impedance layer on the particle surface is insulating at low frequencies, which makes the particles behave as if they are insulating with respect to the solution. At higher frequencies however, capacitive displacement currents can short out this high impedance layer and pass through to the particles, making the particles conductive with respect to the solution. Therefore, the cusp resistance (denoted as R_{cusp} on the left side of Fig. 2), represents the composite resistance without the effect of the high

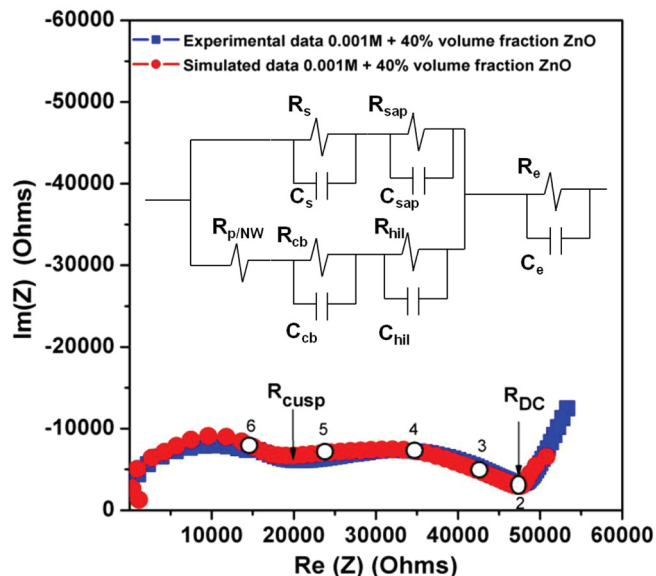


Figure 2. (Color online) Nyquist plot representation of the simulated (red circles) and experimental (blue squares) impedance spectra for a 0.001 M NaCl solution with 40% volume fraction of ZnO ceramic powder. The small numbers along each plot are frequency markers that represent the $\log(\text{frequency})$. Shown as an inset is the equivalent circuit model (adapted from and discussed in detail in Ref. 4) used in the work for simulating PSC impedance spectra, where $R_{p/NW}$ represents the particle or NW resistance, s represents the solution, sap represents solution around the particle or NWs, cb represents current bunching, hil represents the high impedance layer surrounding the particles or NWs, and e represents the electrode.

impedance surface layer and is used for extracting the conductivity of particle. The conductivity calculated from the impedance spectra is thus a sum of both ionic as well as electronic conductivity.⁵

Shown as an inset in Fig. 2, is the equivalent circuit used to simulate the impedance spectra in this work. It is essentially the same as the model proposed by Ingram and Mason and the details are described fully in Ref. 4. In brief, at low measurements frequencies, the resistance of the high impedance layer, R_{hil} , surrounding the particles is high and the bottom branch of the circuit is effectively open. At higher frequencies, the displacement currents short the high impedance layer and current flows through the bottom branch. In order to compensate for non-homogeneities in the real system, the capacitors in this model are replaced by constant phase elements, as explained in Ref. 4.

The PSC method has been demonstrated for measuring the conductivity of insulating and conducting particles of various shapes.^{4-6,18,20,21} A requirement for the PSC method is knowledge about the size and shape of the ceramic powder being analyzed, since this knowledge allows one to choose the appropriate effective medium theories for analyzing the conductivity of the system. The ZnO ceramic powder used in this study is roughly spherical and the model proposed by Bruggeman is suitable⁴

$$\text{Spherical (ceramic powder): } \frac{(\sigma_c/\sigma_s) - (\sigma_p/\sigma_s)}{(\sigma_c/\sigma_s)^{1/3}(1 - (\sigma_p/\sigma_s))} = (1 - f) \quad [1]$$

The ZnO NWs, however, have an approximately ellipsoid shape with a broad range of sizes, and the model proposed by Fricke is used instead^{4,5}

$$\text{Ellipsoid (Wires): } \sigma_p = \sigma_c - \frac{(\sigma_s - \sigma_p)}{1 + \frac{\rho}{3} \sum_{\alpha} \frac{(\sigma_s - \sigma_p)}{\sigma_p - \eta_{\alpha} \sigma_s}} (1 - f) \quad [2]$$

In Eqs. 1 and 2, σ_c , σ_p , and σ_s are the conductivity of the composite, particles or NWs, and solution, respectively; f is the volume fraction of the suspended phase; η_{α} is the form factor which depends on the ellipsoid ratio; and ρ is the density of ZnO.

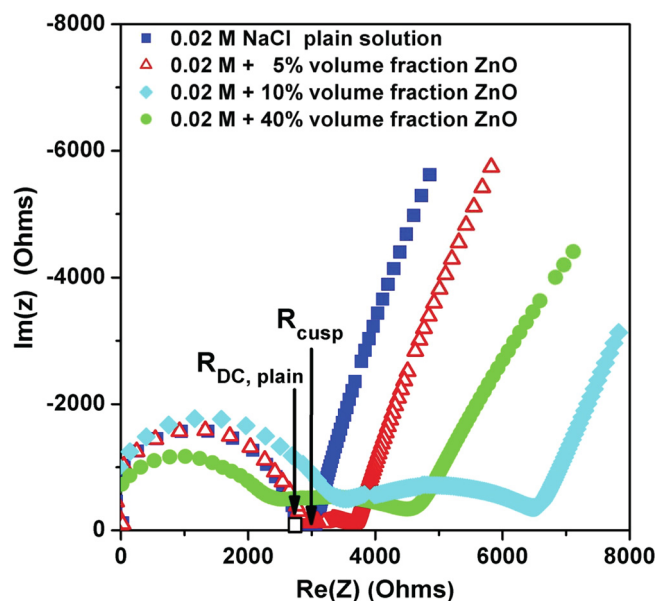


Figure 3. (Color online) Nyquist plot representation of the impedance spectra of a plain 0.02 M NaCl solution (blue squares), and 0.02 M NaCl composite solutions containing either 5% (red triangles), 10% (cyan diamonds), or 40% (green circles) volume fraction of ZnO ceramic powder. The small numbers along each plot represent log (frequency).

Theoretically, the particle or NW conductivity (σ_p) can be calculated directly from Eqs. 1 and 2 when σ_c and σ_s are measured. However, the single point datum can lead to a large error in determining σ_p due to uncertainties in (i) controlling the volume fraction from the loss of some powder/water when the electrodes are squeezed before running each measurement and (ii) reading the resistance from the Nyquist plot for each individual measurement.⁴ These problems can be addressed by performing an experiment over a wide range of σ_s . This is the basis for PSC method. If a series of data can be collected throughout the regime where the solution and particle conductivity are comparable ($\sigma_p \sim \sigma_s$), a crossover point where $\sigma_s = \sigma_p$ (obtained from a plot of $\log[\sigma_s]$ vs. $\log[\sigma_c]$) will be observed.^{4,5} This crossover point allows a more accurate determination of σ_p since at this point, σ_c is equivalent to both σ_p and σ_s ($\sigma_c = \sigma_p = \sigma_s$) and the volume fraction term in Eqs. 1 and 2 will not affect the conductivity.

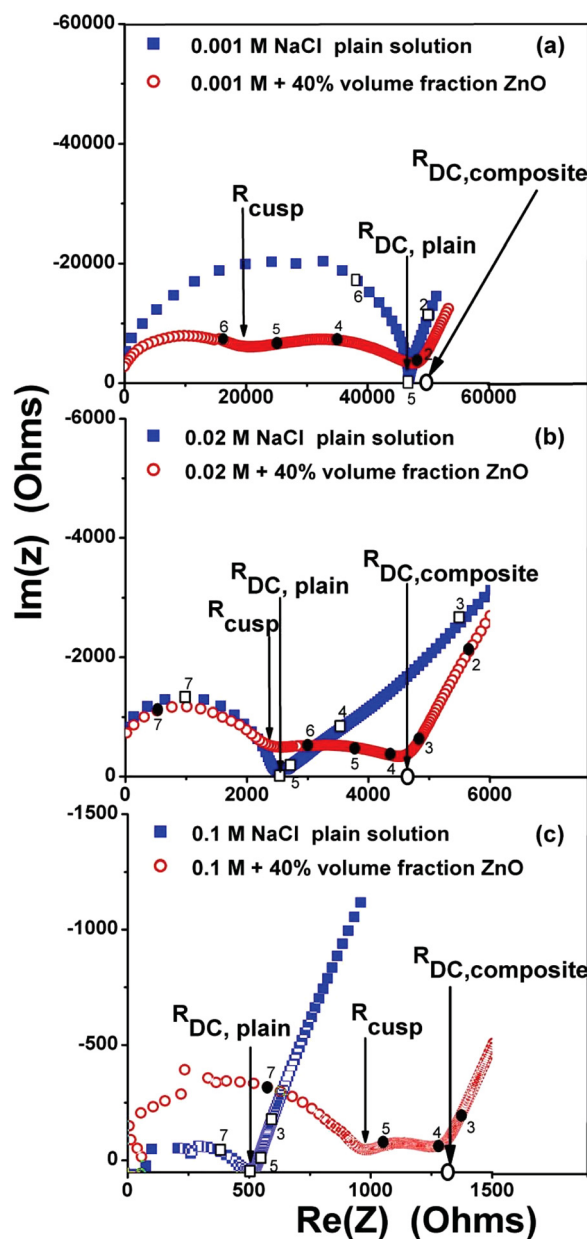


Figure 4. (Color online) Nyquist plot representation of the impedance spectra of 40% volume fraction ZnO powder/solution composites of (a) 0.001 M NaCl, (b) 0.02 M NaCl, and (c) 0.1 M NaCl. The small numbers along each plot represent log (frequency).

A parallel percolation path may exist in the equivalent circuit⁴ in Fig. 2. As discussed in Ref. 22, as long as the conductivity of this particle-particle conduction path is significantly lower than the conductivity of the solution-particle path at the crossover point in a plot of $\log[\sigma_c]$ vs. $\log[\sigma_s]$, it will have a negligible effect on the PSC analysis. Agglomeration or settling of the particles may contribute to percolation. If the degree of agglomeration does not change dramatically from sample to sample, agglomeration should have a negligible effect on the crossover point.²² To minimize any potential impact of agglomeration and ensure uniform dispersal of the nanowires, all nanowire/NaCl solutions were agitated for 2 min prior to each measurement. All impedance measurements (which last approximately 5 min) were performed immediately after this procedure. As a control, a set of measurements that was performed under constant agitation was found give the same results as the measurements performed with agitation prior to the measurement.

Shown in Fig. 3 are impedance spectra of 0.02 M NaCl solutions either plain (without ZnO powder) or mixed with a 5%, 10%, or 40% volume fraction of ZnO powder. (The 0.02 M NaCl concentration was chosen due to its matching conductivity with the bulk ZnO conductivity obtained from four-point probe measurements of sintered ceramic bars.) It is seen that R_{cusp} matches well the solution resistance ($R_{\text{DC, plain}}$) with only a small deviation (<15% error), regardless of the volume fraction of ZnO ceramic powder. The error may be due to the length measurement of the solution in polyethylene tube or small air bubbles within the solution. These results indicate that, at the crossover point, the composite resistance has little dependence on volume fraction, as postulated but not shown in Ref. 4.

Shown in Figs. 4a–4c are AC impedance spectra for both plain NaCl solutions and powder composite solutions (NaCl solution + 40% volume fraction of ZnO ceramic powder) of 0.001, 0.02, and 0.1 M, respectively. The impedance spectra of the plain NaCl solution exhibited two arcs for all NaCl concentrations, confirming the: the left arc represents the bulk component, while the right arc corresponds to the electrode. $R_{\text{DC, plain}}$ is indicated by the x-axis intersection between the electrode and bulk arcs and also corresponds to the DC measurement.⁴

In Fig. 4a, it is seen that R_{cusp} is much lower than $R_{\text{DC, plain}}$, indicating that the particle is much more conductive than the solution ($\sigma_p > \sigma_s$). As NaCl concentration increases, both $R_{\text{DC, plain}}$ and R_{cusp} decrease. Figure 4b shows the nearly perfect coincidence between

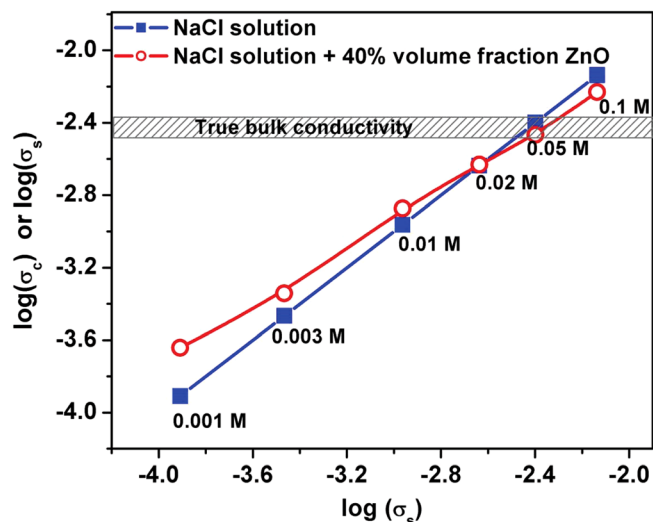


Figure 5. (Color online) Plot of $\log(\sigma_c)$ and $\log(\sigma_s)$ vs. $\log(\sigma_s)$ for a 40% volume fraction ZnO ceramic powder solution (open circles) and a plain NaCl solution (squares), respectively. The crossover point indicates the conductivity of the ZnO powder. The horizontal gray bar, labeled true bulk conductivity, is the conductivity range obtained from four-point probe measurements on ceramic bar samples.

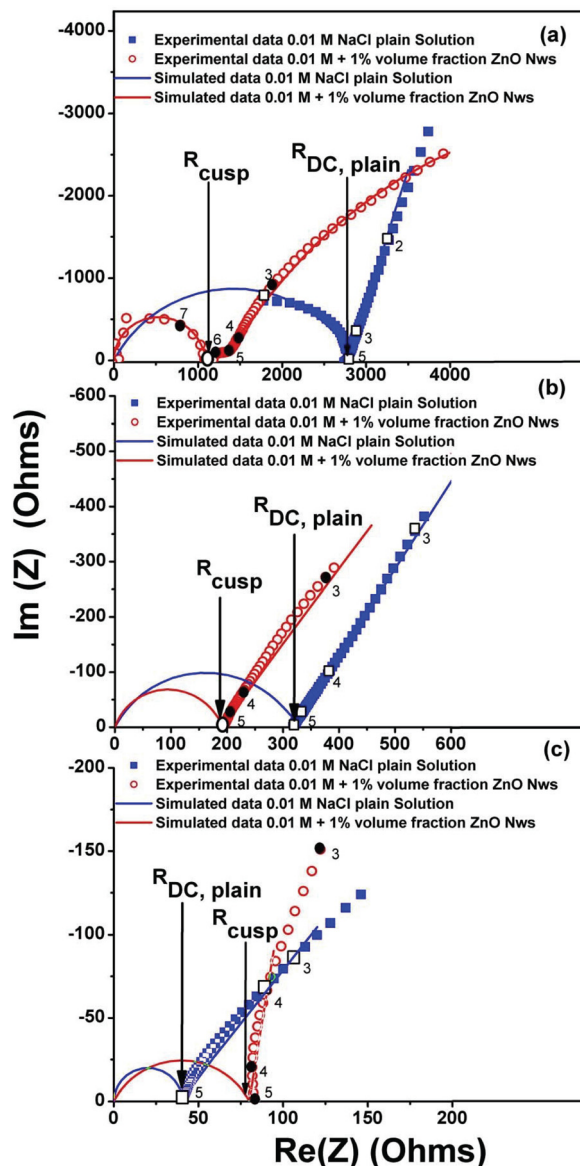


Figure 6. (Color online) Nyquist plot representation of the impedance spectra of plain and 1% ZnO nanowire/solution composites of (a) 0.01 M, (b) 0.1 M, and (c) 1 M NaCl. The solid lines are simulated using the equivalent circuit shown in Fig. 2. The small numbers along each plot represent $\log(\text{frequency})$.

$R_{\text{DC, plain}}$ and R_{cusp} for the 0.02 M NaCl solution and composite solution ($\sigma_p - \sigma_s$), respectively. However, if NaCl concentration increases beyond this point, the solution becomes more conductive than the particle ($\sigma_s > \sigma_p$). This situation is seen in Fig. 4c and is indicated by $R_{\text{DC, plain}} < R_{\text{cusp}}$. In summary, Fig. 4 demonstrates that the set of NaCl solution concentrations covers the regime in which the particle and solution conductivity are comparable.

Shown in Fig. 5 is a plot of $\log(\sigma_c)$ vs. $\log(\sigma_s)$ for each individual experiment from the data in Fig. 4. The crossover point in Fig. 5 yields $\sigma_p = 2.6 \times 10^{-3} \pm 5 \times 10^{-4} \text{ S cm}^{-1}$, which is close to the average sintered bar value of $3.69 \times 10^{-3} \text{ S cm}^{-1}$ determined from our four-point measurements (shown as a gray horizontal bar in Fig. 5). The close match between the conductivity obtained from impedance measurements of ZnO powder and four point probe measurements of a sintered ZnO ceramic bar, suggest that the PSC method can also be a reliable method for measuring the conductivity of NWs.

Shown in Figs. 6a–6c are AC impedance spectra for both plain NaCl solutions and composite ZnO NW solutions (NaCl

Table I. Literature values for conductivity of ZnO NWs

Group	Conductivity (S cm^{-1})	Synthesis method (Precursor)	Selective Growth Agent	Dimension ($l \times d$)
This work	1.2×10^{-2}	Solid vapor (ZnO + C)	Carbonized photoresist	l : 10 μm , d : 45–50 nm
Liu ^a (Ref. 24)	2	Solid vapor (ZnO)	—	d : 60–100 nm
Umar ^a (Ref. 25)	4.1×10^{-5}	Solid vapor (Zn)	—	l : 10–30 μm , d : 60–100 nm
Suh ^a (Ref. 26)	4.3–11.5	Solid vapor (ZnO + C)	Zinc Acetate Dihydrate	l : 5–6 μm , d : 30–70 nm
P-C Chang (Ref. 27)	$(8.8\text{--}119) \times 10^{-2}$	Solid vapor (ZnO + C)	Sn Film	l : 1–3 μm , d : 18–190 nm
Weber (Ref. 3)	1.2×10^{-2}	Solid vapor (Zn)	6H-, 4H-SiC	d : 150 nm
Sakurai ^a (Ref. 28)	2.2	Solid vapor (ZnO + C)	Au	l : 1–50 μm , d : 50–400 nm
Scrymgeour (Ref. 29)	100	Solution based ($\text{Zn}(\text{NO}_3)_2$)	ZnO	l : 4 μm

^aConductivity values calculated from reported mobility and carrier concentration.

solution + 1% volume fraction of ZnO NWs) for NaCl concentrations of 0.01, 0.1, and 1 M, respectively. The solid lines are simulated using the equivalent circuit shown in Fig. 2. A similar trend in impedance spectra is observed for NWs as was seen for the ZnO powder, in which $\sigma_c > \sigma_s$ at low NaCl solution concentration and $\sigma_c < \sigma_s$ at high NaCl solution concentration. For the ZnO NWs, however, the difference between R_{DC} and R_{cusp} is not as clear due to the lower concentration of NWs in NaCl solution. This phenomenon was also observed in the ceramic powder solution in Fig. 3 and in other systems such as carbon nanotube solutions.²³ As the concentration of powder (or NWs) is decreased, the high impedance layer between the matrix (NaCl in this case) and the ZnO powder (NWs) will become insignificant (i.e. NaCl dominates the impedance response). When the NaCl concentration is low, as shown in Fig. 6a, the plain NaCl solution has a higher resistance than the ZnO NWs (judged from the intersection between R_{cusp} and x-axis). When the NaCl concentration is increased, the resistance of the plain NaCl solution becomes lower than the ZnO NWs, as revealed in Fig. 6c. The close fit of the simulation (solid line) to the data shows that the equivalent circuit in Fig. 2 is still a valid description of the system.

As seen in the plot of $\log(\sigma_c)$ vs. $\log(\sigma_s)$ shown in Fig. 7, the crossover point for NW solutions occurs between 0.1 and 0.3 M. In order to observe the impedance difference between the pure and composite solutions clearly, the volume fraction should not be too low. Observation of the crossover point is an indication that the 1% ZnO NW volume fraction is sufficient for the PSC method. From the crossover point, the conductivity of the NWs is estimated to be approximately $1.2 \times 10^{-2} \text{ S cm}^{-1}$. The conductivity of NWs is

strongly dependent upon the growth process. As shown in Table I, the value of conductivity obtained from the PSC method is within the wide range of values reported for ZnO NW devices.

Conclusion

Currently, a major challenge facing the use of NWs is in the wafer scale electrical integration of these quasi-one dimensional structures. A very promising integration technique that has recently emerged is AC dielectrophoresis. In dielectrophoresis, NWs suspended in solution are flowed over the surface of the device wafer and AC electric fields are used to align the NWs between lithographically defined electrodes.⁸ Before dielectrophoresis can be fully realized in a production environment, a non-destructive method of rapidly assessing the quality of batches of NW solution, prior to use on a device substrate, will be needed. The powder-solution-composite (PSC) technique was introduced by Ingram et al.⁴ as a method of using AC impedance spectroscopy to determine the conductivity of ceramic powders in solution. In this work, we have adapted the PSC method to characterize the conductivity of ZnO NWs in NaCl solution. We find that the PSC method is not dependent on particle volume fraction and allows in-situ measurements of NW solutions. We demonstrate that the PSC technique offers a non-destructive measurement platform for measuring the average conductivity of large quantities of NWs without forming electrical contacts to the NWs.

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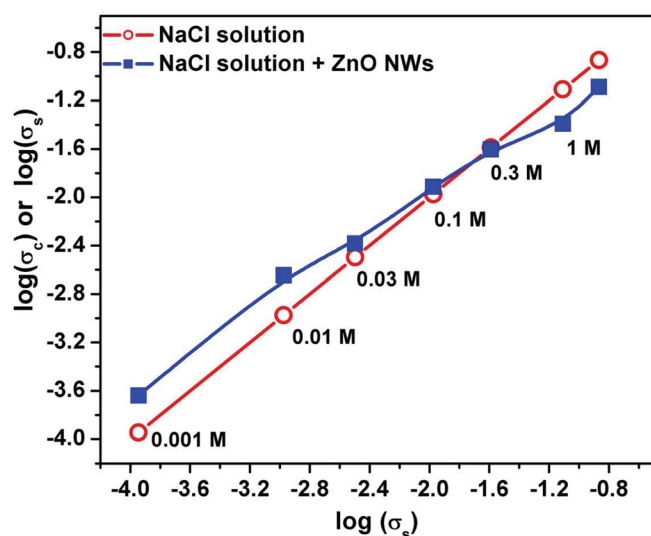


Figure 7. (Color online) Plot of $\log(\sigma_c)$ and $\log(\sigma_s)$ vs. $\log(\sigma_s)$ for plain NaCl solutions (squares) and ZnO NW (open circles) NaCl solutions, respectively. The crossover point indicates the conductivity of the ZnO NWs.

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